

Designation: D8175 – 18

Standard Test Method for Finite Flash Point Determination of Liquid Wastes by Pensky-Martens Closed Cup Tester¹

This standard is issued under the fixed designation D8175; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

This flash point test method is a dynamic test method that depends on specified rates of heating to be able to meet the precision of the test method. The rate of heating may not in all cases give the precision quoted in the test method because of the low thermal conductivity of some materials.

Flash point values are a function of the apparatus design, the condition of the apparatus used, and the operational procedure carried out. Flash point can, therefore, only be defined in terms of a standard test method, and no general valid correlation can be guaranteed between results obtained by different test methods or with different test apparatus.

This test method, and Test Method D8174, are used to determine the flash point of liquid wastes. These procedures are primarily derived from Procedures A and B of Test Methods D93 (EN ISO 2719), and are informally known as Pensky-Martens.

1. Scope

1.1 This test method covers the procedure for a finite flash point test, within the range of 20 to 70 °C, of liquid wastes using a manual or automated Pensky-Martens closed cup tester.

1.2 This test method contains two procedures and is applicable to liquid waste, liquid phase(s) of multi-phase waste, liquid waste with suspended solids, or liquid waste that tends to form a surface film under test conditions.

Note 1—If the liquid waste is of a viscosity such that the subsample volume will not be uniformly heated under the test conditions even with the increased stir rate of Procedure B, then use the small-scale method (Test Method D8174 for Finite Flash Point Determination of Liquid Wastes by Small-Scale Closed Cup Tester).

1.3 Procedure A is applicable to non-viscous liquids that are without suspended solids. Procedure B is applicable to viscous liquids, liquids with suspended solids, or liquids that form films.

Note 2—This test method is not applicable for corrosive liquid wastes (see Test Method D8174).

1.4 *Units*—The values given in SI units are to be regarded as the standard.

1.5 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the

responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use. Warning statements appear throughout. Also see applicable Safety Data Sheets (SDS) for information about certified reference materials (CRMs) or secondary working standards (SWSs) that may be used in the analysis. SDS may also be useful if some components of the waste sample are known.

1.6 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

2. Referenced Documents

- 2.1 ASTM Standards:²
- D93 Test Methods for Flash Point by Pensky-Martens Closed Cup Tester
- D6299 Practice for Applying Statistical Quality Assurance and Control Charting Techniques to Evaluate Analytical Measurement System Performance
- D8174 Test Method for Finite Flash Point Determination of Liquid Wastes by Small-Scale Closed Cup Tester

¹ This test method is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.01.06 on Analytical Methods.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

- E502 Test Method for Selection and Use of ASTM Standards for the Determination of Flash Point of Chemicals by Closed Cup Methods
- E1137/E1137M Specification for Industrial Platinum Resistance Thermometers
- E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids
- 2.2 ISO Standards:³
- ISO 17034 General Requirements for the Competence of Reference Material Producers
- ISO Guide 35 Reference Materials—General and Statistical Principles for Certification
- ISO 2719 Determination of Flash Point—Pensky-Martens Closed Cup Method
- ISO 60751 Industrial Platinum Resistance Thermometers and Platinum Temperature Sensors

3. Terminology

3.1 Definitions:

3.1.1 *ambient temperature, n—in waste flash point test methods,* the temperature in the immediate surroundings where the flash point apparatus is located.

3.1.2 *dynamic, adj*—condition in which the vapor above the subsample and the subsample are not in temperature equilibrium at the time that the ignition source is applied.

3.1.2.1 *Discussion*—This is primarily caused by the heating of the subsample at the constant prescribed rate with the vapor temperature lagging behind the subsample temperature.

3.1.3 *flash point*, *n*—*in waste flash point test methods*, the lowest temperature of the subsample, adjusted to account for the variation in atmospheric pressure from 101.3 kPa, at which the application of an ignition source causes the vapors of the subsample to ignite under the specified conditions of the test.

3.1.4 safety check, n—in waste flash point test methods, the initial dip of the ignitor into the test cup with subsample, before the heating process begins, to check if the sample has a flash point at or below 20 °C.

4. Summary of Test Method

4.1 A brass test cup of specified dimensions, filled to the inside mark with a waste subsample and fitted with a cover of specified dimensions, is heated and the subsample stirred at specified rates, using one of two defined procedures, A or B. An ignition source is directed into the test cup at regular intervals with simultaneous interruption of the stirring, until a flash is detected. The flash point is reported as defined in 3.1.3.

5. Significance and Use

5.1 This procedure is intended to be used to evaluate the ignitability of liquid wastes.

5.2 Flash point measures the response of the subsample to applied heat and an ignition source under controlled laboratory

conditions. It is only one of a number of properties that must be considered in assessing the overall flammability hazard of a liquid waste material.

5.3 Flash point can indicate the possible presence of highly volatile and flammable materials in a relatively nonvolatile or nonflammable material.

6. Interferences

6.1 Metals such as brass can react with corrosive wastes to give off hydrogen gas, which can cause a false positive flash. Regardless of film formation, all corrosive wastes shall be analyzed using a small-scale tester with a stainless steel cup (Test Method D8174).

6.2 Halogenated Constituents:

6.2.1 The presence of some halogenated constituents in the waste may cause the flash to appear green instead of blue.

6.2.2 Some halogenated compounds can flash and some halogenated compounds only burn. The burning of a halogenated constituent within the waste should not be confused with a flash. See Appendix X1 for additional information.

6.3 Because of the nature of the waste itself, the flash point results of the waste can be inconsistent (greater than reproducibility). An example would include volatile droplets suspended in a gel. Additional testing may be required to determine the lowest detectable flash point.

7. Apparatus

7.1 Pensky-Martens Closed Cup Apparatus (Manual)—This apparatus consists of the test cup, cover and shutter, stirring device, heating source, ignition source, temperature measuring device, air bath, and top plate described in detail in Annex A1 and Annex A2. The assembled manual apparatus, test cup, cover, and test cup assembly are illustrated in Figs. A1.1-A1.4, respectively. Dimensions are listed respectively.

7.2 Pensky-Martens Closed Cup Apparatus (Automated)— This apparatus is an automated flash point instrument that is capable of performing the test in accordance with Section 12 of this test method. The apparatus shall use the test cup, cover and shutter, stirring device, heating source, ignition source, and temperature measuring device described in detail in Annex A1 and Annex A2.

7.3 *Draft Shield*—A shield located at the back and on two sides of the instrument, for use in circumstances in which protection from drafts does not exist.

7.4 *Barometer*—With accuracy of 0.5 kPa or better. Barometers that have been pre-corrected for use at weather stations or airports are not suitable.

8. Reagents and Materials

8.1 *Cleaning Solvent*—Use a solvent suitable for cleaning out the subsample from the test cup. Two commonly used solvents are toluene and acetone. **Warning**—Toluene, acetone, and many solvents are flammable and a health hazard.

8.2 *Butane, Propane, and Natural Gas*—For use as a pilot and ignition source (not required if an electric ignitor is used). **Warning**—Butane, propane, and natural gases are flammable and a health hazard.

³ Available from International Organization for Standardization (ISO), ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, http://www.iso.org.

8.3 Reference Materials—See Annex A3.

9. Sampling

9.1 Sampling should address the intended use of the analysis and should include representative subsampling from the source waste material. When possible, obtain at least a 250-mL sample from the source waste material for single-phase wastes. If multiple-phase waste is suspected, obtain a greater volume of sample to ensure adequate phase volumes for testing. Approximately 75 mL of subsample is used for each test in a finite flash point determination.

9.2 Samples that have multiple phases (layers) shall be separated into single phases. Each separate liquid phase is then tested.

9.3 Store samples in clean, tightly sealed containers at normal room temperature (20 to $25 \,^{\circ}$ C) or colder. Avoid freezing the sample. If the sample is frozen, allow the sample to warm such that it is a liquid. Do not store samples for an extended period of time in gas-permeable containers such as plastic, because volatile material can diffuse through the walls of the container. Samples in leaky containers that can lose volatiles are suspect and may not provide valid results.

Note 3—If a regulatory decision point is being assessed for a waste in a leaky container, the lack of a flash point or a flash point exceeding the regulatory decision point may not satisfy the regulatory assessment.

9.4 Erroneously high flash points can be obtained when precautions are not taken to avoid loss of volatile materials. Do not open containers unnecessarily.

10. Preparation of Apparatus

10.1 Place the apparatus on a level, steady surface. Tests are to be performed in a draft-free room or compartment. Tests made in a ventilation hood with active airflow or in any location where drafts occur may not be reliable. Use a draft shield (7.3) when necessary.

Note 4—For samples with noxious vapors, it is permissible to place the apparatus with a draft shield in a ventilation hood. The draft may be adjusted so that vapors can be withdrawn without causing air currents over the test cup during the ignition source application period.

10.2 Read the manufacturer's instructions on the care and servicing of the apparatus and for the correct operation of any controls.

10.3 Prepare the apparatus for operation in accordance with the manufacturer's instructions for calibrating, checking, and operating the equipment. **Warning**—An incorrectly set test flame size or incorrect positioning for an electric ignitor can significantly affect the test result.

10.4 Clean the test cup, cover, and its accessories with an appropriate cleaning solvent (8.1) to remove any traces of gum

or residue from the previous test. Wipe dry with absorbent paper. A stream of dry, clean air may be used to remove the last traces of solvent. Dispose of solvents and waste material in accordance with local regulations.

10.5 If not automatically recorded by the instrument, measure and record the ambient atmospheric pressure using the barometer (7.4) at the time of each test.

10.6 An electronic thermal flash detector may be used in lieu of a visual observation of the flash. In cases of dispute, the visual observation shall be used.

11. Verification of Apparatus

11.1 Verify and correct, if necessary, the readings on the temperature measuring device (A2.1 and A2.2) at least every twelve months according to the manufacturer's instructions, and that the temperature measuring device is in accordance with Annex A2.

11.2 Verify the performance of the apparatus before initial use and at least once per year by determining the flash point of a CRM. Examples of suitable liquids, and their approximate flash points, are listed in Annex A3. Use a CRM that has a flash point that is reasonably close to the expected temperature range of the samples to be tested. The flash point of the reference material shall be tested in accordance with Sections 12 and 13. The flash point obtained shall be within the limits stated in Table A3.1 for the identified CRM, or within the limits calculated for an unlisted CRM (Annex A3).

11.3 Once the performance of the apparatus has been verified, the flash point of secondary working standards (SWSs) can be determined along with their control limits. These secondary materials can then be used for more frequent performance checks (see Annex A3).

211.4 When the flash point obtained is not within the limits stated in 11.2, check the condition and operation of the apparatus to ensure conformity with the details listed in Annex A1, especially with regard to tightness of the cover (A1.1.2.1), the action of the shutter, the position of the ignition source (A1.1.3), and the angle and position of the temperature measuring device. After any adjustment, repeat the test in 11.2 using a fresh subsample, with special attention to the procedural details prescribed in this test method.

11.5 The numerical values obtained during the verification check (11.2) shall not be used to provide a bias statement, nor shall they be used to make any correction, such as to the temperature calibration of the apparatus or to the flash points subsequently determined using the apparatus.

FINITE FLASH POINT DETERMINATION

12. Procedure

PROCEDURE A

12.1 Non-Viscous Liquids That Are Without Suspended Solids:

12.1.1 Due to the limited temperature range of this test, all start temperatures for the subsample and test cup shall be at 20 \pm 5 °C.

Note 5—If ambient temperature is greater than 20 °C, refrigeration of the subsample and test cup may be required to obtain the starting temperature. It is acceptable for the sample temperature to be lower than the start temperature of the test at the commencement of the test.

12.1.2 Ensure that the sample is representative of the waste form being tested (9.1). Fill the test cup with the subsample to the filling mark inside of the test cup. If too much subsample has been added to the test cup, remove the excess using a syringe or similar device for withdrawal of fluid. Place the cover on the test cup and place the assembly into the apparatus. Be sure the locating or locking device is properly engaged. If the temperature measuring device is not already in place, insert the device into its holder.

12.1.3 Light the test flame and adjust it to a diameter of 3.2 to 4.8 mm, or switch on the electric igniter and adjust the intensity in accordance with the manufacturer's instructions.

Warning—Gas pressure should not be allowed to exceed 3 kPa.

Warning—Exercise care when using a gas test flame. If it should be extinguished, it will not ignite the vapors in the test cup, and the gas from the test flame that then enters the vapor space can influence testing.

Warning—The operator should exercise caution during the initial application of the ignition source, since subsamples containing low-flash material can give an abnormally strong flash when the ignition source is first applied.

Warning—The operator should exercise caution during the performance of this test method. The temperatures attained during this test method, up to 70 $^{\circ}$ C, can cause burns.

Warning—As a safety practice, it is strongly advised, before heating the test cup and subsample, to dip the ignitor to check for the presence of unexpected volatile material.

12.1.4 Apply the heat to the test cup, as indicated by the temperature measuring device, at such a rate that the temperature increases 2.5 to 3.5 °C/min.

Note 6—The ramp rate control may be difficult to achieve at the low end of the testing range.

12.1.5 Turn on the stirring device at 90 to 120 rpm, stirring in a downward direction.

NOTE 7—Meticulous attention to all details relating to the ignition source, size of test flame or intensity of the electric ignitor, rate of temperature increase, and rate of dipping the ignition source into the vapor of the test subsample is necessary for good results.

12.1.6 When the subsample, sample cup, and temperature measuring device have been installed, apply the ignition source as a safety check (manually or using an automated function of the instrument) and then each time the temperature of the

subsample increases by increments of 1 °C. Discontinue the stirring of the subsample and apply the ignition source by operating the mechanism on the cover that controls the shutter so that the ignition source is lowered into the vapor space of the cup in 0.5 s, left in its lowered position for 1 s, and quickly raised to its upward position. If a flash is detected, proceed to 12.1.7. If no flash is detected, proceed to 12.1.10.

Note 8—If the starting temperature is well above the actual flash point, the upper explosion limit could be exceeded because the volume of volatiles generated could displace the oxygen and no flash response would be detected. If this condition is suspected, then use Test Method D8174 and suitable apparatus for below-ambient-temperature flash point testing.

12.1.7 Record the detected flash point (from the reading on the temperature measuring device) and the ambient barometric pressure at the time the ignition source application causes a flash in the interior of the test cup. The temperature should be recorded to the nearest 1 °C. The sample has flashed when a large flame appears and instantaneously propagates itself over the entire surface of the subsample. Occasionally, particularly near the actual flash point, the application of the ignition source can cause a blue halo or an enlarged flame; this is not a flash and should be ignored.

Warning—For certain mixtures containing halogenated hydrocarbons, such as methylene chloride or trichloroethylene, no flash, as defined, is observed. Instead a significant enlargement of the test flame (not halo effect) and change in color of the test flame from blue to yellowish-orange occurs. Continued heating and testing of these samples above ambient temperature can result in significant burning of vapors outside the test cup and can be a potential fire hazard. See Appendix X1 and Appendix X2 for more information. Other halogenated compounds, such as 1,2-dichloroethane, can cause the test flame or the flash to appear green instead of blue.

12.1.8 When the apparatus has cooled down to a safe handling temperature, remove the cover and the test cup and clean the apparatus as recommended by the manufacturer.

Note 9—Exercise care when cleaning and positioning the lid assembly so as not to damage or dislocate the flash detection system or temperature measuring device. See the manufacturer's instructions for proper care and maintenance.

12.1.9 Prepare a fresh subsample and repeat the test. If a flash is detected on the second safety check, record the barometric pressure and temperature. Report the flash point result as less than 20 °C. If the flash point is 20 °C or below, consider retesting the sample using the small-scale tester (Test Method D8174).

12.1.10 Begin application of temperature ramp as described in 12.1.4 and turn on the stirring motor as described in 12.1.5. Continue the application of the ignition source as described in 12.1.6 for every 1 °C until a flash is observed or until 70 °C is reached. When a flash is observed, record the information as described in 12.1.7 and proceed to 12.1.11. If no flash is observed up to 70 °C, proceed to 12.1.12.

12.1.11 Clean the system. Prepare a fresh subsample and repeat the test with the subsample and the test cup at 5 $^{\circ}$ C

lower than the detected flash (but not lower than 20 °C). Repeat the test with additional subsamples if needed (refer to 12.1.13). Proceed to Section 13 for reporting of flash point results.

12.1.12 Clean the system. Prepare a fresh subsample and repeat the test with the subsample and the test cup at 70 $^{\circ}$ C.

12.1.12.1 When a flash is detected on a fresh subsample at 70 °C, it may indicate that loss of low concentrations of vapors during frequent testing prevented the detection of a flash point during the temperature ramp from 20 to 70 °C. Therefore, the test must be repeated on fresh subsamples over 10 °C ranges until the finite flash point is detected. A duplicate test will need to be performed to confirm the finite flash point. Repeat the test with additional subsamples if needed (refer to 12.1.13). Proceed to Section 13 for reporting of flash point results.

12.1.12.2 If no flash is detected, report the flash point as greater than 70 $^{\circ}\mathrm{C}.$

12.1.13 Two detected results obtained within the repeatability precision in Section 15 are acceptable. If the repeatability precision for the two results are not acceptable by Section 15, repeat the testing on one or more fresh subsamples. If results between three or more tests are inconsistent, then use the lowest corrected flash point temperature in place of the average corrected flash point temperature.

PROCEDURE B

12.2 Viscous Liquids, Liquids with Suspended Solids, or Liquids That Form Films:

12.2.1 Proceed as prescribed in 12.1, with the exception of the requirements for rates of stirring and heating.

12.2.2 Turn on the stirring device at 250 ± 10 rpm, stirring in a downward direction.

12.2.3 Apply the heat at such a rate that the temperature as indicated by the temperature measuring device increases 1 to $1.5 \,^{\circ}C/min$.

13. Calculation rds. iteh. ai/catalog/standards/sist/920e25b

13.1 *Pressure Correction Calculation*—If the ambient barometric pressure (10.5) differs from 101.3 kPa, correct the detected flash point using Eq 1 as follows:

Corrected Flash Point =
$$C + 0.25(101.3 - A)$$
 (1)

where:

C = detected flash point, °C, and

A = ambient barometric pressure, kPa.

13.2 Average Corrected Finite Flash Point Result Calculation—Average two or more corrected results for the final reported flash point of a sample:

Average Corrected Finite FP Result:
$$\bar{x} = \frac{1}{n}(x_1 + ... + x_n)$$
 (2)

where:

n = number of subsample tests,

 \bar{x} = average finite flash point result, and

 x_n = corrected subsample flash point result.

Note 10—Supplemental information for non-SI units can be found in Appendix X3.

14. Report

14.1 Report the corrected finite flash point average rounded to the nearest 1 $^{\circ}$ C.

14.1.1 If required, convert the flash point to degrees Fahrenheit using Eq 3:

((Corrected Flash Point, in Celcius) \times 1.8) + 32

14.2 Report the test method and procedure (A or B) used, the identification of the material tested, the test date, and any deviation, by agreement or not, from the procedures specified in Section 12.

14.3 Because of the nature of some waste materials, it may be difficult to determine two or more corrected flash points within repeatability. If the waste flashes over a range of temperatures, the lowest flash point shall be reported (see 12.1.13). Additionally, if adequate for the use of the data, a "less than" result may be reported.

15. Precision and Bias

15.1 *Precision*—It is not possible to specify the precision of the procedure in Test Method D8175 for measuring flash point because, by its nature, every sample of liquid waste is compositionally unique and therefore it is not meaningful to provide a precision statement. Limited work to demonstrate repeatability precision for some surrogate liquid wastes is shown in Appendix X4.

15.2 *Bias*—The procedure in this test method has no bias because flash point can be defined only in terms of this test method.

16. Keywords

16.1 automated flash point; automated Pensky-Martens closed cup; combustible; fire risk; flammability; flash point; ignitability; liquid waste; Pensky-Martens closed cup; volatile



ANNEXES

(Mandatory Information)

A1. APPARATUS SPECIFICATIONS

A1.1 A typical assembly of the apparatus, gas heated, is shown in Fig. A1.1. The apparatus shall consist of a test cup, cover, and stove conforming to the following requirements:

A1.1.1 *Cup*—The cup shall be of brass and shall conform to the dimensional requirements in Fig. A1.2. The flange shall be equipped with devices for locating the position of the cup in the

stove. A handle attached to the flange of the cup is a desirable accessory. The handle shall not be so heavy as to tip over the empty cup.

A1.1.2 Cover:

A1.1.2.1 *Cover Proper*—The cover shown in Fig. A1.3 shall be of brass (A1.1.1) and shall have a rim projecting downward



NOTE 1—Lid assembly can be positioned either right or left-handed.

FIG. A1.1 Pensky-Martens Closed Cup Tester